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PRODUCTION OF A GLASS FIBER WITH HIGH TENSILE STRENGTH

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Claims

1. Method for the production of a quartz glass fiber with a high tensile strength by drawing a quartz glass preform, characterized in that a layer which inhibits crystallization of silicon oxide to tridymite and/or cristobalite on the surface of the preform is first applied on the preform.

2. Method according to Claim 1, characterized in that for the production of the layer, boron oxide or boron oxide and silicon oxide are used.

The invention concerns a method for the production of a quartz glass fiber with high tensile strength by drawing a quartz glass preform.

In communication engineering, the optical transmission of information by means of communication cables made of hair-thin optical fibers is increasingly gaining in importance. In cabling [structures] and in the laying of cables, the fibers are exposed to a high tensile load. They must therefore have high tensile strength.

These fibers are preferably produced in that a quartz glass preform, for example, a cylindrical or hollow-cylindrical rod is conducted through an inductive heating zone or another drawing furnace and thereby drawn out. The preform can, for example, be produced in the manner described in "Siemens Forschungs- und Entwicklungsberichte" [Siemen's Research and Development Reports], 5 (1976), pages 171-175. Immediately thereafter, the fibers are coated, in a corresponding molten bath, with a layer, preferably made of plastic, for protection from mechanical damage.

For the tensile strength, an upper limit of approximately  $18,000 \text{ N/mm}^2$  results from the binding energy of the Si-O bond. In fact, this limit itself is not approximately reached even with the best fibers. Thus, the peak values for fibers with a length of 20 m in laboratory samples are only approximately  $10,000 \text{ N/mm}^2$ . In mass production, only values of approximately  $1000 \text{ N/mm}^2$  are obtained.

In the journal "Siemens Forschungs- und Entwicklungsberichte" [Siemen's Research and Development Reports], 7 (1978), pages 158-165, the following are given as causes for the tearing of the fibers: dust particles from the atmosphere or microfissures on the surface of the preform, crystalline occlusions in the glass matrix, impurities or particles, which are drawn in during the drawing of the fibers and arrive at the surface of the fibers, and damage of the fiber surface, which arises during the coating or winding of the drawn fibers and during the laying of insufficiently coated fibers.

In order to rule out these sources of error to a large extent, the surfaces of the preforms are first carefully chemically cleaned and, immediately before the drawing, freed from absorption substances, in particular, condensed moisture, by flame polishing, wherein, at the same time, scratches or fissures on the surfaces are completely restored. Then, at approximately  $2000^\circ\text{C}$ , the preforms are drawn to form fibers and, immediately after leaving the drawing furnace, coated, in a bath, with the protective layer, which is dried in a downstream furnace. A multiple coating and drawing of the fibers can be provided, before they are wound onto a storage drum.

From the cleaning of the preform to the leaving of the coating baths, the fibers must be conducted through a dust-free atmosphere, which is preferably produced by the blowing in of an inert gas. In order to avoid the formation of microfissures in the drawn fibers these precautionary measures, however, are not sufficient.

The goal of the invention is to make possible, by suitable measures, the drawing of fibers that have a higher tensile strength.

The invention is based on the knowledge that with the heating of the preform in the drawing furnace, large numbers of small tridymite and cristobalite crystals are formed on the surface of the quartz glass preform during the drawing ; they are not melted quantitatively in the drawing furnace. With the subsequent cooling of the fibers below 275°C, these crystals flake off from the surface of the fibers and leave behind a microfissure. Small cristobalite crystals on the surface of the preform can be detected to a considerable extent by X-ray investigations. In part, they may be melted if the residence time of the preform in the drawing furnace is correspondingly long, but the working rate is thereby considerably reduced.

The invention is also based on the additional knowledge that not only the drawn-in dust particles, which act as crystallization nuclei, but rather, in particular, the silicon oxide's own nuclei are also regarded as the formation cause of these small crystals. At high fiber-drawing temperatures (approximately 2000°C), a considerable part of the silicon oxide is already evaporated. A fraction of this vapor condenses on the surface of the preform and there acts as a nucleus for the crystallization of tridymite (at temperatures above 1100°C) and cristobalite (temperatures above 1400°C). With the traditional furnace constructions and the usual fiber-drawing rates (several meters per second), a preform stays, on the average, approximately 1-2 h, at temperatures above 1100°C. In this time, the crystallization nuclei grow to form grains, whose size can be several 100 µm.

This invention extensively eliminates these previously unknown causes of fiber fissures. This occurs in that a layer is first applied on the preform in a prepared work step; this layer inhibits the formation of tridymite and/or cristobalite crystals on the surface of the preform during the drawing . Such a layer can preferably be made of boron oxide ( $B_2O_3$ ) or boron oxide and silicon oxide.

A boron oxide layer can be advantageously produced by deposition from a corresponding gas phase. As a gas phase, a gaseous mixture of boron chloride ( $BCl_3$ ) and oxygen are used. The preform, however, can also be produced by immersion into a corresponding boron oxide melt ( $B_2O_3$ ). Since the vapor pressure of the boron oxide is only approximately 0.2 torr at fiber-drawing temperatures (approximately 2000°C), it is sufficient if this layer has a thickness of only a few µm.

Advantageously, the boron oxide from the boron oxide layer can be diffused into the preform at temperatures at which the surface of the preform is in a liquid-viscous state. In this way,  $B_2O_3/SiO_2$  surface layers are obtained, which also prevent the formation of nuclei and the crystallization of tridymite and cristobalite and--assuming very thin  $B_2O_3$  coats--have sufficient thickness. The preform has a liquid-viscous surface in various stages of the method, for example, during the flame polishing or during immersion into a correspondingly hot boron oxide melt.

A boron oxide-silicon oxide layer can also be applied in that the preform is immersed into a melt of these two oxides; however, the layer can also be produced by deposition from a gas phase containing boron, oxygen, and silicon (for example,  $\text{BCl}_3$ ,  $\text{SiCl}_4$ ,  $\text{O}_2$ ). The boron can be introduced deeper into the preform by diffusion from this layer also.

The layer, which can contain other crystallization-inhibiting additives (for example,  $\text{Al}_2\text{O}_3$ ) instead of boron oxide, protects the surface of the preform from having the silicon oxide, which evaporates in the drawing zone and is condensed out on the preform, act as a crystallization nucleus for cristobalite and/or tridymite during the drawing of the fibers. In this way, the formation of microfissures during the subsequent cooling of the drawn fibers is largely prevented so that fewer demands can be made on the quality of the protective layer which is applied at the end. Thus, for example, plastics which are cheaper, easier to apply, and easier to cure can be used for this protective layer. At the same time, the residence time of the preform can be reduced in the drawing zone so that as a whole, higher drawing rates and a more economical production can be attained.

The invention is explained in more detail with the aid of two figures and an exemplified embodiment.

Figure 1 shows an apparatus to apply the layer on the surface of a preform for optical fibers, which are made of quartz glass and have a refractive index which is higher in the fiber core than in the fiber jacket, by the addition of foreign atoms.

The needed preform is produced, in a known manner, according to the chemical vapor deposition method, by coating a glass tube on the inside with corresponding quartz glass layers by deposition from a corresponding gas phase. Subsequently, the glass tube is collapsed to form a cylindrical preform.

In the apparatus according to Figure 1, the preform 1 is held in the chuck 4 of a turning lathe. The deposition of the protective layer from the gas phase is undertaken by movement of the housing 2 of a flame device that can be moved along the arrow 3.

To this end, a first burner 5 is provided in the housing, to which, in accordance with arrows 6 and 7,  $\text{SiCl}_4$  and  $\text{BCl}_3$  or  $\text{O}_2$  are supplied. This burner is surrounded by a water cooling unit 8. From the flame directed at the tube, an  $\text{SiO}_2/\text{B}_2\text{O}_3$  protective layer is deposited, whose thickness is determined by the throughput of the gas mixture and the advance rate of the burner.

The deposited protective layer is melted to a clear, solid film by the flame of an oxyhydrogen gas burner 9, located next to the layer. 10 and 11 show the supply conduits for hydrogen and oxygen to the oxyhydrogen gas burner 9. The waste gases of this burner are conducted into a fume hood in accordance with the arrow 12.

Simultaneous with the application of the protective layer, the glass tube is softened by the heat from the burner. With this liquid-viscous state of the preform, the deposited boron oxide

diffuses into deeper layers, so that a  $B_2O_3$ - $SiO_2$  protective layer, approximately 5  $\mu m$  in thickness, is formed on the preform surface. The preform produced in this manner can be drawn out to form fibers, for example, in the manner described in the aforementioned reference "Siemens Forschungs- und Entwicklungsberichte."

According to Figure 2, the coating of a preform, for example, of a cylindrical, quartz glass body 20, which was described above and was produced according to the CVD method, is carried out by immersion in a  $B_2O_3$  melt 21, which is maintained [as a] liquid by a heating spiral 24, coiled around the melting crucible 23. For the immersion in accordance with the arrows 25, only a short immersion time is needed, which is sufficient enough so as to ensure a uniform wetting of the formed body. A thickness of the wetting film of approximately 2-5  $\mu m$  is sufficient to protect the surface of the crystallite formation.

2 Patent claims

2 Figures

### Summary

In order to obtain a fiber with a high tensile strength during the drawing of a quartz preform, for example, an optical fiber for the optical transmission of information, a layer is first applied on the preform; this layer inhibits a crystallization of silicon oxide to tridymite and/or cristobalite on the surface of the preform. Silicon oxide, which evaporates in the drawing furnace and precipitates on the colder preform, is not able to act then as a crystallization nucleus for tridymite or cristobalite. Such crystals, which are not melted in the drawing furnace, could namely flake off from the surface of the fiber and leave behind fissures (Figure 1).

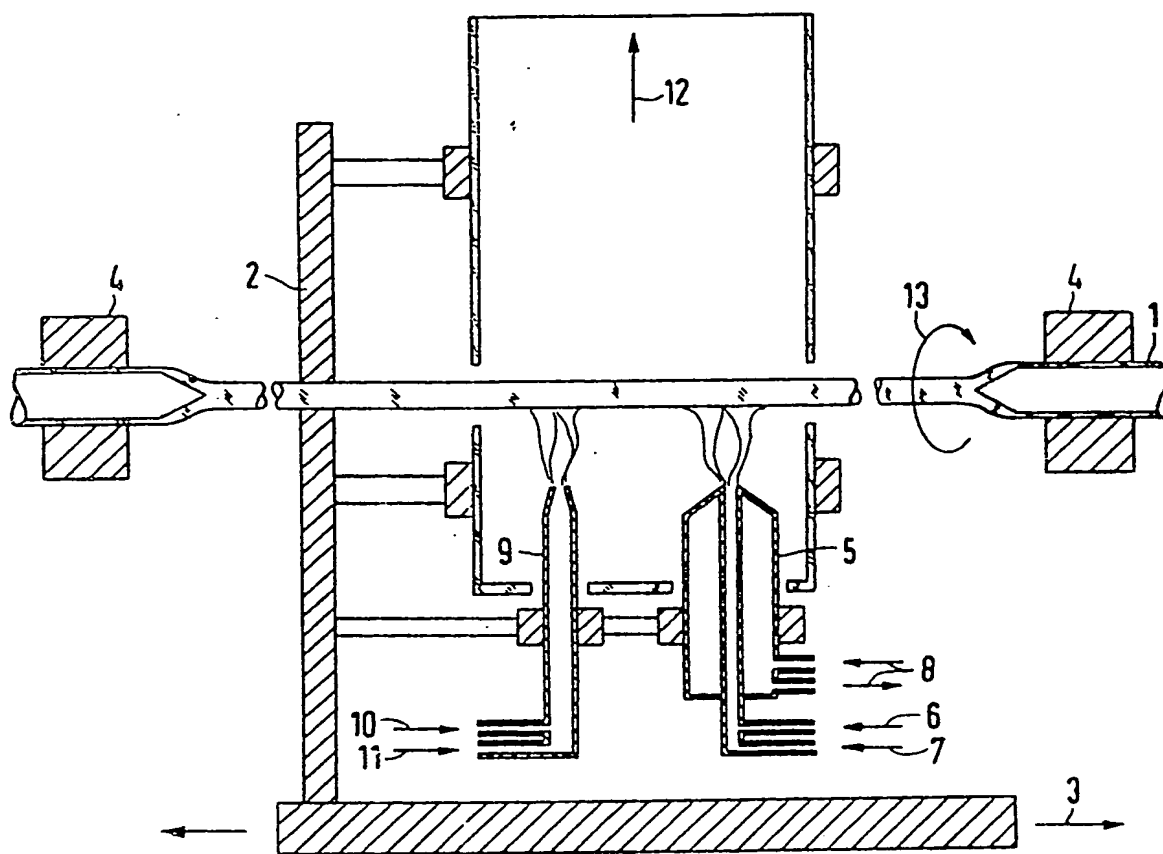


FIG 1

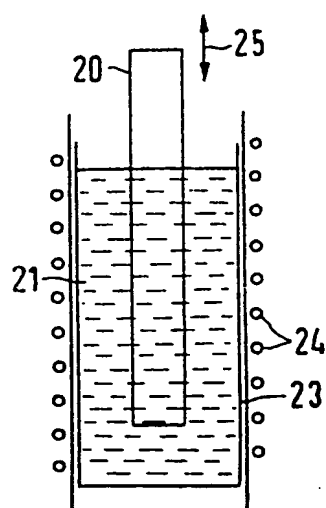


FIG 2